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Homogenization kinetics of a nickel-based superalloy produced by powder bed fusion laser sintering

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ABSTRACT

Additively manufactured (AM) metal components often exhibit fine dendritic microstructures and elemental segregation due to the initial rapid solidification and subsequent melting and cooling during the build process, which without homogenization would adversely affect materials performance. In this letter, we report *in situ* observation of the homogenization kinetics of an AM nickel-based superalloy using synchrotron small angle X-ray scattering. The identified kinetic time scale is in good agreement with thermodynamic diffusion simulation predictions using microstructural dimensions acquired by *ex situ* scanning electron microscopy. These findings could serve as a recipe for predicting, observing, and validating homogenization treatments in AM materials.

Additive manufacturing (AM) is a suite of emerging technologies that creates three-dimensional metal [1], polymer [2], or ceramic [3] objects directly from digital models through an additive process.

While the potential of metal-based AM has been clearly demonstrated [4,5], significant challenges remain from the perspective of materials science and engineering. In particular, metal-based AM processes such as selective laser melting [6], direct metal laser sintering [7], and electron beam melting [1] often involve rapid heating and cooling of metal powders to and from material melting temperatures. Combined with the additive nature of the build processes, heterogeneous microstructure, residual stress distortion, and material inconsistency are difficult to avoid [8]. These characteristics adversely affect the materials performance. To fulfill the potential of AM, these challenges must be addressed.

To minimize such problems, post-build heat treatments of AM parts are necessary to create a homogenized microstructure with reproducible mechanical properties. While the *ex situ* effect of post-build heat treatment has been evaluated for AM materials [9–11], an *in situ* study that reveals the fundamentally important heat-treatment kinetics remains elusive. In this *letter*, we present a study that combines modeling prediction, *in situ* high-energy synchrotron X-ray scattering experiments, and *ex situ* microscopic characterization to elucidate the homogenization kinetics in AM-produced Inconel 625 (IN625). In particular, we introduce a novel *in situ* SAXS characterization scheme that follows the diffusion (homogenization) process of a fixed sample volume at elevated temperature, and we present a correlation-coefficient analysis that quantifies the homogenization kinetics and reveals the temporal evolution of the microstructures.

IN625 is a nickel-based superalloy primarily strengthened by the solid-solution hardening from niobium and molybdenum in a nickel-chromium matrix. IN625 features a combination of high yield strength, fatigue strength, and excellent oxidation, creep and corrosion resistance in aggressive environments. IN625 is widely used in the industries where complex shapes and consequent extensive machining are often required. Due to this reason, AM was recognized as an appealing option for fabricating IN625 components during the early stage of AM development [9,12]. Improved understanding of the detailed microstructural changes during the post-build heat treatment not only provides opportunities to optimize the mechanical properties of AM IN625 components, but also sheds light on the more general behavior of AM alloy components.

Table 1 shows the manufacturer-certified chemical composition of the raw powder material used in this study. A sieve analysis showed that 98% of the powder particles have diameters <80 μm. The as-built IN625 specimen was produced using an EOS M270 laser-sintering powder-bed fusion system (EOS GmbH, Munich, Germany).[1] The laser was operated at 195 W with a scanning speed of 800 mm/s and a hatch

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[1] Certain commercial equipment, instruments, software or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the Department of Commerce or the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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Table 1
Chemical compositions (mass fraction × 100) of the raw IN625 powder (from EOS [20]), and in the interdendritic and dendritic regions of the as-built sample, and after a 1 h, 1150 °C heat treatment.

	Ni	Cr	Mo	Nb	Fe	Ti	Al	Co
IN625 Powder	65.44	20.11	8.79	4.03	0.74	0.35	0.34	0.20
As-built Interdendritic regions[a]	65.75 ± 0.32	25.05 ± 0.08	5.36 ± 0.20	2.21 ± 0.10	0.38 ± 0.02	0.16 ± 0.02	0.33 ± 0.04	0.11 ± 0.02
As-built On the dendrites[a]	61.94 ± 1.38	24.18 ± 0.41	6.87 ± 0.47	4.41 ± 0.99	0.34 ± 0.03	0.20 ± 0.03	0.60 ± 0.14	0.09 ± 0.03
1150 °C, 1 h heat treated[b]	63.74 ± 0.02	21.10 ± 0.00	8.32 ± 0.02	4.00 ± 0.01	0.71 ± 0.00	0.36 ± 0.00	0.32 ± 0.00	0.21 ± 0.00

[a] EDS analyses performed on etched samples.
[b] Uncalibrated values.

spacing of 100 μm. The test blocks were removed from the base plate by electro-discharge machining without a stress-relieving heat treatment. Specimens were cut and mechanically polished following standard metallographic procedure [13].

The optimal temperature for homogenization heat treatment was predicted using the CALPHAD-based Thermotech Ni7 [14] thermodynamic database and the NIST Ni-based atomic mobility database [15]. Diffusion simulations at 870 °C and 1150 °C were performed using the DICTRA software [16], which is a finite difference code that assumes local equilibrium at each grid point and then solves a flux balance equation at each grid point for each time step.

The microstructure and elemental spatial distributions of the as-built and homogenized samples were obtained using a JEOL S-7100F field-emission scanning electron microscope (SEM) and energy dispersion spectroscopy (EDS). The SEM specimens were oriented in the plane perpendicular to the base plate which allows microstructural information related to the dendrite centers, inter-dendritic regions, and intra-dendritic regions to be acquired. To investigate the homogenization kinetics, we performed *in situ* synchrotron small-angle X-ray scattering (SAXS) experiments using the ultra-small angle X-ray scattering instrument at the Advanced Photon Source, Argonne National Laboratory [17,18]. The SAXS sample was treated following a two-step heat treatment. The first annealing step at 870 °C for 1 h is the recommended stress-relieving heat treatment for IN625 [19]; the second anneal at 1150 °C for 1 h is the homogenization treatment called for by thermodynamic modeling, as described later. More experimental details can be found in the Supplementary materials.

Fig. 1(a) shows the representative microstructure of the as-built IN625 sample. It can be seen from Fig. 1(a) that the cellular/dendritic microstructure manifests itself as both a columnar structure and an equiaxed cellular structure, depending on the angle between the solidification direction and the viewing plane. Both types of segregated domains have been extensively observed in and ubiquitously associated with AM metal components [1,20–23]. These formations are closely tied to the rapid solidification inherent to the selective laser or electron beam melting involved in the AM process. It has been shown that when the melt pool solidifies, rapid cooling can occur *via* conduction into the substrate and the deposit, leading to directional growth of the columnar grains counter to the heat flux direction [9]. We found that the characteristic spacing is slightly different for the columnar dendrites (1 μm to 2 μm) and the cellular dendrites (2 μm to 3 μm), which may be attributed to the differences in the local cooling rates associated with the growth of these different microstructures. These dimensions are similar to those previously identified in AM Ni-based super alloys [9,20,23–26].

Fig. 1(b) shows the EDS elemental maps of Ni, Cr, Nb, and Mo within the same cellular regions of the as-built sample. We observe clear

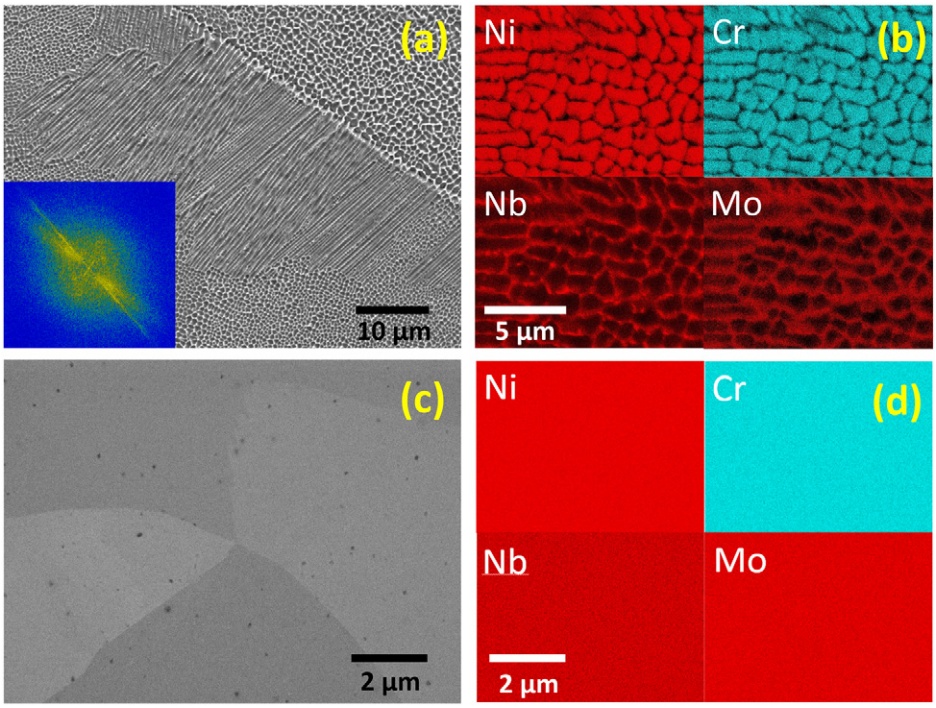

Fig. 1. (a), (b) SE micrograph and elemental X-ray maps of as-built IN625 sample. Inset of (a) shows the Fourier transform of the imaged microstructure. (c), (d): SE micrograph and elemental X-ray maps of 1150 °C, 1 h heat-treated sample.